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LEAK TESTING OF HIGH PRESSURE OXYGEN AND
NITROGEN TANK SYSTEMS FOR THE SKYLAB AIRLOCK*

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ABSTRACT

An existing Propulsion Test facility was modified for use as a leak test facility for acceptance testing of the 3000 psig oxygen and nitrogen gas storage tank systems used on the Skylab Airlock U-1 and U-2 Vehicles. Leak rates in the range of 10^{-2} to 10^{-5} standard cc/sec of helium were measured remotely from an explosion containment test cell. Selection of the test site was based on consideration of techniques, equipment, potential test hazards, leak measurement methods, and the availability of existing applicable equipment from prior test programs. Facility design, operating experience, and test results are discussed.

INTRODUCTION

The life support atmosphere of the Skylab Orbital Workshop is supplied by six oxygen and six nitrogen tank assemblies mounted on the Airlock. Each tank is pressurized to 3000 psig with gas prior to launch. The maximum allowable equivalent helium leak rate at 3000 psig for each tank assembly, including its valve package, is 10^{-2} standard cc/sec.

Although the tanks themselves were leak checked by their suppliers prior to delivery to MDC, additional hydrostatic and leak testing was required after installation of the valve packages and associated pressure transducers at MDC. The tanks were received at MDAC-E and the valve package attached by brazing. The brazed joint connection was designed to permit replacement of a defective valve package without danger of damaging the tank or valve package. The leak test program was designed to assure that the entire tank assembly, including its valve package, was ready for installation on the Airlock vehicle without the necessity for subsequent leak checking.

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Although numerous smaller high pressure tanks had been pressure and leak tested in previous programs, none of them presented as much damage potential in the event of tank or valve package failure. Therefore, special precautions were taken to reduce the risk of harm to personnel and facilities.

TEST SPECIMEN DESCRIPTION AND SAFETY CONSIDERATIONS

The oxygen tanks are constructed with a 321 stainless steel liner reinforced by a filament wound fiberglass-epoxy shell bonded to the liner. The nominal external dimensions are 45 in. diameter and 90 in. long. Minimum internal volume is 57 ft³. The tank assembly weight does not exceed 2500 pounds. Nitrogen tanks are fabricated from titanium hemispheres welded together at their circumference. The nominal external diameter of each spherical nitrogen tank is 41 in., and the minimum internal volume is 19.28 ft³; tank assembly weight did not exceed 393 pounds. The tanks and their control valve packages were hydrostatically tested, prior to leak measurement, with Freon PCA (Precision Cleaning Agent) to 1.50 times the 3000 psig working pressure (or approximately 4500 psig). The nitrogen tank design pressure considerations were 3000 psig working, 5000 psig proof, and 6660 psig burst at 70°F. The oxygen tank assembly design pressure considerations were 4500 psig working, 7530 psig proof and 10,000 psig burst.

Freon PCA was used for three reasons: (1) to avoid potential tank weakening which can occur if, for example, methyl alcohol is used to hydrostatically test titanium tanks at high pressure, (2) to avoid potential corrosion problems of valve packages when water is used, and (3) to permit thorough removal of the hydrostatic test fluid after testing. The nitrogen tanks have an operating temperature range of -20 to 160°F. The oxygen tanks have a similar temperature restriction but with an additional requirement to avoid temperature changes in the liner of 1°F per minute or more, which might cause debonding of the tank liner from its reinforcing filament wound fiberglass-epoxy reinforcement.

An initial examination of the potential hazards of leak testing large high pressure tanks revealed that it would be unwise to conduct the work in a conventional building. The types of tanks to be tested had exhibited excellent safety records in previous programs and the likelihood of tank rupture was considered very slight. However, tank rupture due to flaws, human error, or any other cause could produce catastrophic results in a conventional laboratory. For example, calculations indicated that the 19.28 ft³ nitrogen tank at 3000 psig contained potential energy equivalent to 14.0 pounds of TNT. The 57 ft³ oxygen tank at 3000 psig contained the energy of approximately 41.0 pounds of TNT, although, due to the type of tank construction, the energy might be released at a slightly less explosive rate.

The need to perform the leak tests in a remote safe facility became evident. A high pressure fuel test facility with a 7 ft diameter vacuum chamber became available from another program. The chamber was located in a test cell designed for explosion containment. Cell operations could be monitored and controlled from an adjacent control room. A study showed that the facility could be modified economically within the available schedule time.

The test cell chosen had been designed to withstand an internal pressure 225 psi above normal atmospheric pressure without rupturing the walls and without forcing the doors open. Calculations indicated the possibility that in the event of a tank failure, shrapnel could acquire enough energy to damage but not penetrate the foot-thick cell walls which were constructed of concrete with two independent grids of 1-1/4 in. diameter steel rods on 9 in. centers. The cell doors, made of steel I-beams with welded steel facing plates and filled with concrete, were secured with massive locking plates at their top and bottom. Part of the control cell was separated from the test cell by two reinforced concrete walls, one on each side of an access hallway. The most conservative calculations indicated the possibility that impact of large pieces of shrapnel with the inside surface of the test cell wall might cause spalling of concrete from the opposite side of the wall. Therefore, a barrier of sand bags was erected parallel to the single wall separating a portion of the test cell from the control cell.

SURVEY OF LEAK TEST METHODS

Various ways were considered to measure leak rates of the tank systems. The chamber rate of pressure rise method with the chamber vacuum pump operating was found to be too insensitive and unsteady to measure 10^{-2} standard cc/sec of gas leakage over any reasonable time span. Chamber leakage, chamber outgassing, test article outgassing, chamber temperature changes, and tank system temperature changes (principally heating during the filling process) would have obscured the measurement of the small allowable leakage.

Comparison of tank leakage into a chamber with leakage from a reference manometer was found to be theoretically sensitive enough, but would have been subject to some of the same uncertainties as for the chamber rate of rise method. In both cases, the source of the gas could not be determined to be actual tank system leakage instead of a combination of leakage and outgassing.

A variation of the chamber rate of pressure rise was considered in which the chamber would be evacuated and then sealed off from its pump. Its rate of rise due to leakage, outgassing, etc., would be measured and plotted as a function of time. Then the tank system would be pressurized and the new chamber rate of pressure rise would be plotted. The difference in the slopes of the curves would be interpreted as tank system leakage. This

method would not distinguish accurately between actual tank leakage and outgassing due to tank heating.

Various techniques of bagging the tank system to collect helium leaking from the tank system were considered, but were found to be too insensitive and uncertain for good quantitative results. Helium permeation through the bag and helium nonuniformities within the bag would have been difficult to determine.

A technique was considered in which the tank system would be enclosed within an inner chamber containing its own ion gauge. The inner chamber would be within a conventional space chamber with its ion gauge. The inner chamber would have a gas inleak system and a solenoid valve between the inner and outer chambers. During initial evacuation the solenoid valve would be open to equalize pressures in both chambers; then the solenoid valve would be closed. During a leak test, the rate of rise of the inner chamber relative to the outer chamber would be a measure of the tank system leakage. A measured flow of gas from the inleak system would be used for reference purposes to calibrate the leak rate.

A somewhat similar system using two ion gauges in series, with a passage of known conductance between them, could be used to measure the gas flow from a test article in the inner chamber to the outer chamber. This principle is used in calibrating ultrahigh vacuum gauges by extrapolation of the response of two standard gauges operating within their normal ranges.

All of the methods mentioned above in this section have various drawbacks, particularly the inability to distinguish between true tank system leakage and gas flow from other sources such as outgassing of tank and chamber materials.

The method selected for the leak test program was that of placing the tank system in a vacuum chamber, pressurizing the tank with a mixture of 10% helium and 90% nitrogen, and then measuring the helium leaking into the chamber.

A further description of various leak detection methods used in the Space Systems Laboratory at MDC has been presented elsewhere.¹

FACILITY DESIGN REQUIREMENTS AND ASSEMBLY

A portion of the propulsion test facility which was available for modification for leak testing is shown in plan view in Figure 1. The 7 ft diameter vacuum chamber in Cell 8, normally evacuated only by the mechanical pumps and blowers in the adjacent vacuum system room, was modified to provide high vacuum and leak measurement capability.

The tank system specifications required that each tank, including valve package and pressure and temperature transducers, have an equivalent helium leak rate no greater than 10^{-2} standard cc/sec. With the concurrence of the Airlock Project, a mixture of helium and nitrogen was chosen for leak testing; this was considered acceptable provided that the total gas pressure was 3000 psig and that proper allowance was made for the ratio of helium to nitrogen. Since actual leakage through passages of fixed

geometry was of interest, and since permeation due to diffusion was not a factor for the test articles, a gas mixture was considered to give results substantially as accurate as could be obtained with pure helium. The accuracy and reliability were of primary concern and were established by calibration prior to the test of each tank system. In order to obtain leak rate measurements greater than and less than the allowable maximum of 10^{-2} standard cc/sec, the leak measurement system was designed to measure from less than 10^{-5} to 10^{-2} standard cc/sec equivalent leakage of 100 percent helium. The actual gas mixture used was 10% helium and 90% nitrogen; thus, the leak detector response was an order of magnitude less than it would have been with pure helium. The system was calibrated with a 10^{-3} standard cc/sec leak using 100% helium. Thus the system could be adjusted to measure the maximum allowable leak without encountering detector saturation problems. The system background level was also determined and taken into account to provide for full-range calibration.

The modified system is shown schematically in Figure 2. A 32-in. diameter oil diffusion pump and its associated water-cooled elbow and liquid-nitrogen cooled anti-backstreaming baffle, reclaimed from another deactivated chamber, were added to provide chamber pressures in the 10^{-5} to 10^{-6} torr range. DC-704 silicone pump fluid was used in the diffusion pump. A 130-cfm mechanical pump was used to back the diffusion pump during initial chamber evacuation and during leak detection measurements when the total gas flow from the chamber exceeded the throughput capability of the leak detector. Remote-controlled motorized valves were added to permit the leak detector to share as much as possible of the gas load from the chamber for maximum sensitivity. The original leak detector installation involved separating an NCR series 925-50 leak detector into its modular components. These components included a sensing section (cold trap, diffusion pump, ionizer, collector, etc.), which was installed in the test cell, and a control and instrumentation electronics section, which was installed in the control room. The leak detector was backed by a 15 cfm foreline pump in the test cell. To provide additional protection of the leak detector from contamination, an external liquid nitrogen cold trap was installed between it and the chamber system foreline. All system controls requiring operation during the test were located in the control room or non-hazardous areas for safety.

The liquid nitrogen system which serviced the facility cold traps is shown schematically in Figure 3. The internal cold trap inside the leak detector was filled manually prior to pressurization of a tank system and did not require refilling during a normal test. The other cold traps were kept filled automatically by remote-controlled liquid level sensors.

The helium and nitrogen gas pressurization system is shown in Figure 4. This system permitted the tank system to be pressurized to 300 psig with helium from gas cylinders and to the

full 3000 psig test pressure by the addition of gaseous nitrogen produced by vaporization of high pressure liquid nitrogen.

The vacuum chamber and leak detector combination was provided with a positive means to accurately calibrate the response of the entire system to known helium flow rates. This calibration was in addition to the normal leak detector tuneup and calibration procedures, and was based on fundamental primary standards. The calibration system is shown schematically in Figure 5. Later, in the test program, a second independently operated Veeco helium mass spectrometer leak detector was added to the system to verify the results obtained with the original leak detector and to act as a spare in case of instrument malfunction. The system calibrator located on the chamber was used to calibrate both leak detectors simultaneously.

Prior to the first tank system leak test, the facility was checked out to see that all equipment functioned as planned, and training sessions were conducted to familiarize test personnel with equipment and procedures. A detailed operating test procedure was prepared which included normal operations, emergency procedures, safety precautions, checkoff lists, etc.

TEST OPERATIONS

Prior to a test run, the calibration of all instrumentation was certified as up to date and the proper functioning of the test equipment was verified.

Installation of an oxygen tank system is shown in Figure 6. A transportation dolly was used to bring the tank to the chamber; the tank then was raised to the chamber level and the dolly rails were aligned with mating rails inside the chamber. Installation of a nitrogen tank system is shown in Figure 7. The quilted fiberglass blanket on the nitrogen tank was removed prior to leak testing. The nitrogen tank valve package with pressure transducers is mounted on the front boss of the tank.

Thermocouples were taped to the nitrogen tank to provide additional information during the pressurization and depressurization process. Temperature sensors were attached to the oxygen tank during assembly by the manufacturer.

After installation of a tank system in the chamber, all gas lines inside the chamber were checked for leaks, using a sniffer probe attached to a leak detector. Prior to "sniffing", the lines were pressurized first to 2000 psig with helium, and the pressure was increased to 3000 psig with nitrogen. These tests were conducted with the tank system manual fill valve closed to avoid pressurizing the tank. If no leaks were detected, the chamber was closed and evacuated. The chamber was evacuated with the auxiliary pumping system which consisted of two 500-cfm mechanical pumps and two Roots-type blowers. Approximately twenty minutes were required to attain 5×10^{-2} torr in the chamber.

Next the diffusion pump elbow was cooled with water and the anti-backstreaming baffle in the 32-in. diameter ducting above the diffusion pump was cooled with liquid nitrogen from a 500-gallon insulated trailer. Another 500-gallon trailer supplied a liquid-to-gas converter to produce the 3000-psig gaseous nitrogen necessary to pressurize the tank systems being tested.

When chamber pressure reached 5×10^{-5} torr, or less, the auxiliary pumping system was closed off and the 130 cfm pump, in conjunction with the 32-in. diffusion pump was used for system calibration and leakage measurement. Next the leak detectors were tuned individually with their helium sensitivity calibrators.

The chamber leak calibration system, shown in an earlier sketch (see Figure 5), was used to permit a calibrated flow of helium to enter the chamber. The motorized valves between the chamber foreline, the NRC leak detector inlet, and the 130-cfm backing pump were adjusted to provide a steady high response on both leak detectors. At least three calibration runs were made to assure calibration stability. The leak calibration system used a Veeco pinched tube to regulate the helium flow to 3×10^{-3} scc/sec. The value was verified each time with the inclined manometer.

After system calibration, the tank system was slowly pressurized to 300 psig with helium then increased in 500 psig steps to a maximum of 3000 psig with nitrogen. The 3000 psig pressure was maintained for at least one hour to permit pressure and temperature stabilization to occur and to ensure reliable leak rate measurements.

One of the test requirements was to measure tank fill valve seat leakage with the tank at 3000 psig, after the one-hour stabilization period. No suitable remote procedure was devised for this, since the valve had to be closed manually and the torque measured during closing. Hence, with the approval of safety personnel, the chamber was backfilled to ambient pressure, and the required test personnel entered the test cell to connect a bubble burette to a vent valve outside the chamber. Personnel left the area while the leakage gas, if any, was being collected. The maximum allowable leakage past the valve seat was 13.7 standard cc/hr. At this time, a quick helium sniffer test was performed to locate any leaks which might have been indicated by the tank system leak test described previously.

After the high pressure test was completed, tank pressure was reduced to 435 psig and the flow rate of gas through the tank system vent valve was measured with a flowmeter. The minimum allowable flow rate was 15.1 standard cfm. The tank gas pressure was then reduced to 100 psig, and a sample of gas collected for analysis of any trace of Freon PCA which might have been left in from the hydrostatic testing which preceded the gas leak test. The tank system was then removed from the chamber for return to the Airlock assembly areas. The tank systems exhibiting any anomalies were refurbished and retested until all specification requirements were met.

TEST RESULTS

For the U-1 Skylab Airlock Vehicle, six nitrogen and six oxygen tank assemblies were tested. Likewise, for the U-2 Vehicle, six nitrogen and six oxygen tanks were tested. Some of the leakage tests disclosed anomalies, causing rework and retesting. However, all successfully met specification requirements before being delivered for assembly into their respective vehicles. A summary of tank assembly test results is presented in Table 1.

The facility functioned satisfactorily from the first tank system leak test in May 1971 to the most recent test in March 1973.

SUMMARY

An existing propulsion test facility was modified for use as a leak test facility for acceptance testing of the 3000 psig oxygen and nitrogen gas storage tank systems used on the Skylab Airlock U-1 and U-2 Vehicles. Quantitative leak rates in the range of 10^{-2} to 10^{-5} standard cc/sec of helium were measured remotely from an explosion containment test cell. The program, which covered the period of May 1971 through March 1973, was successful in testing all 13 nitrogen and all 13 oxygen tank assemblies to assure that they are flight ready.

REFERENCES

- ¹ McKinney, H. F., "Practical Applications of Leak Detection Methods," the Journal of Vacuum Science and Technology, Vol. 6, No. 6, Nov/Dec, pages 958-964.

ACKNOWLEDGEMENTS

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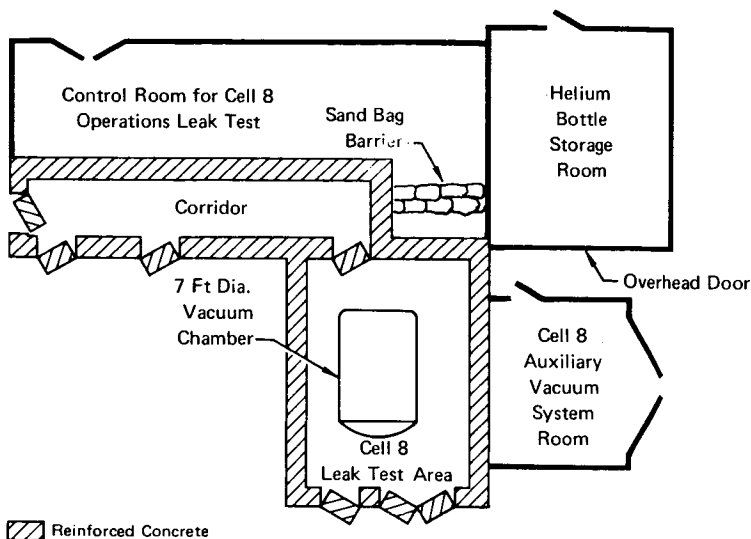


Figure 1 - Leak Test Facility

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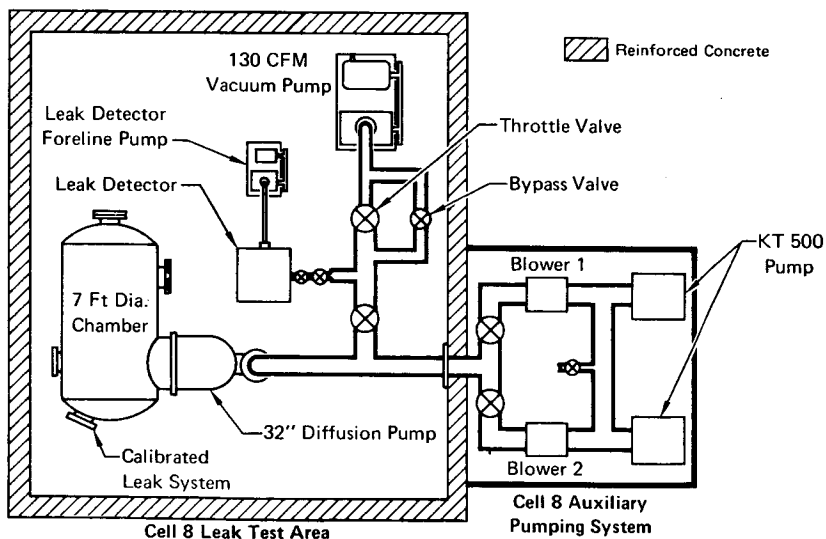


Figure 2 - Seven Foot Diameter Vacuum Chamber System Modified to Provide High Vacuum and Leak Measurement Capability

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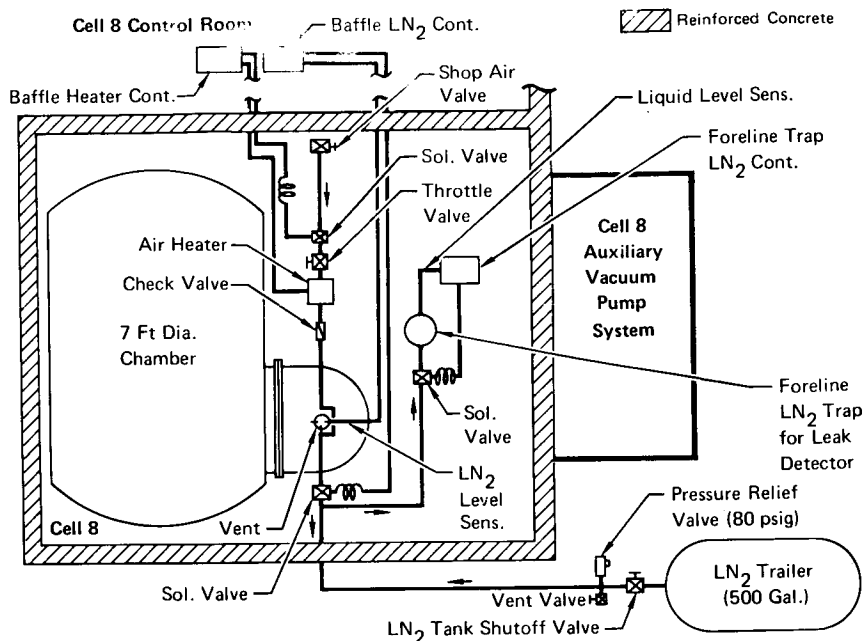


Figure 3 - Seven Foot Diameter Vacuum Chamber Liquid Nitrogen System GP73-3324 3

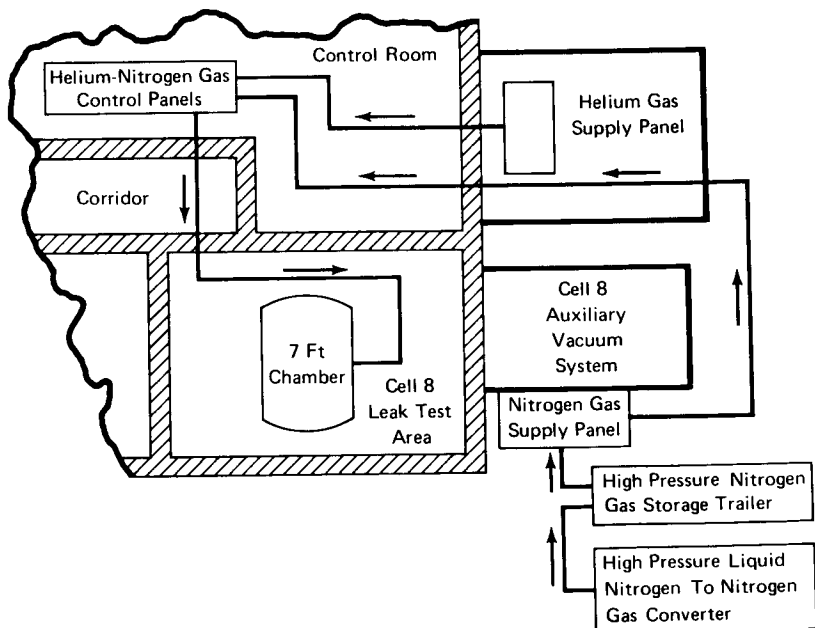


Figure 4 - N₂-He Gas Pressurization System GP73-3324 4

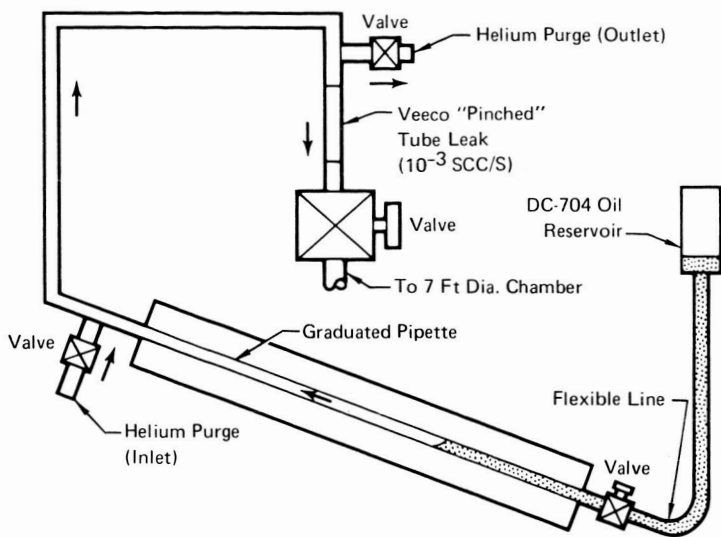


Figure 5 - Chamber Leak Calibration System

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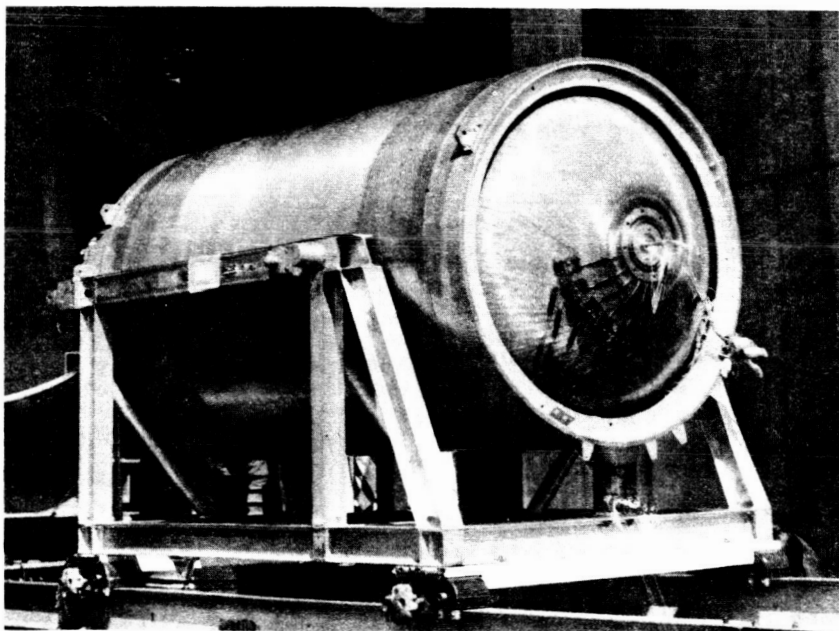


Figure 6 - Oxygen Tank System in Position in Front of 7 Foot Chamber of Cell 8

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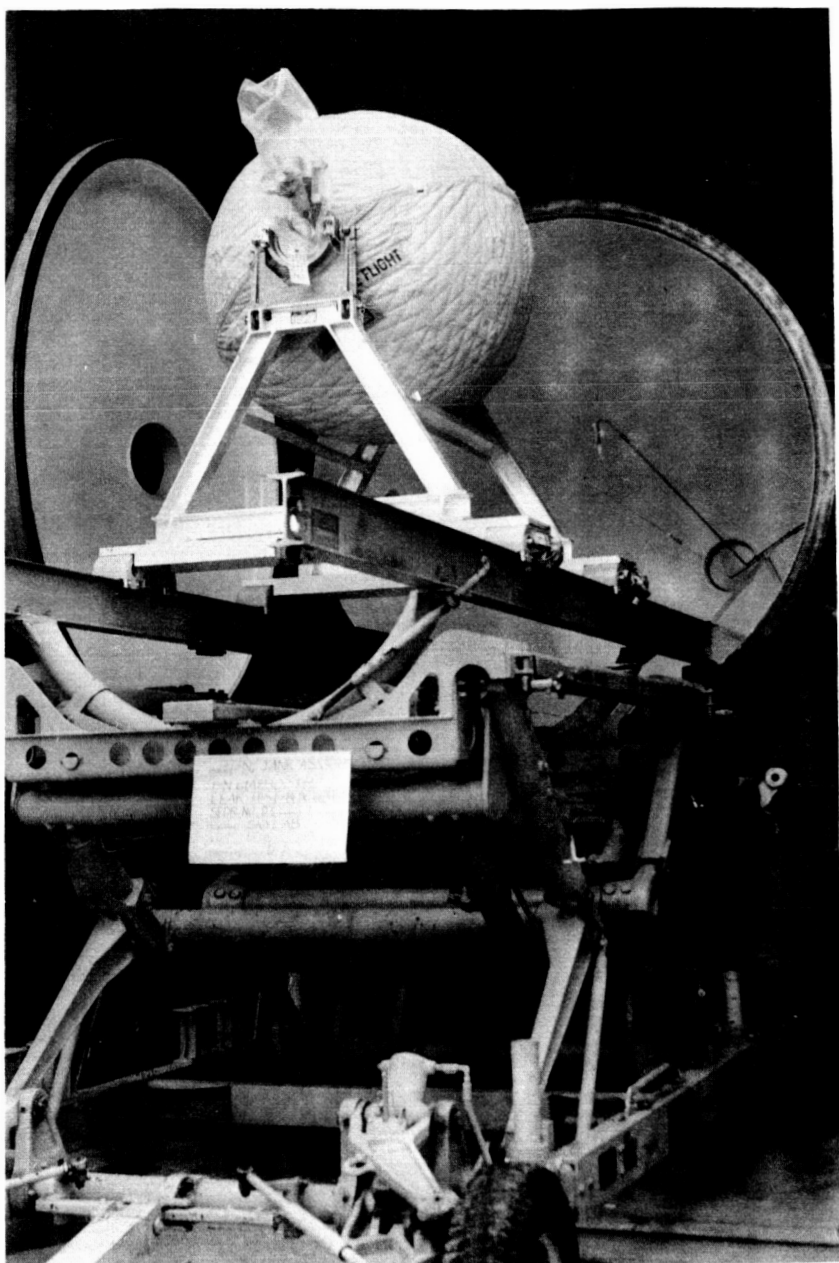


Figure 7 - Nitrogen Tank Prior to Installation in Vacuum Chamber
Quilted Fiberglass Blanket was Removed Before Leak Testing

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Table 1 - O₂ - N₂ Tank Test Data

Vehicle	S/N	MDC P/N	Type of Tank	Test No. Test Chamber Pressure	MRR	Test Date	Assembly Leak Rate SCC/Sec (10% H ₂)	Fill Valve Seat Leakage SCC/HR 10% He, 90% N ₂	Vent Valve Flow Rate SCF/MIN 10% He, 90% N ₂
U-1	0003	61A830248	N ₂	1/Vac 2/Amb	A51AE8	5-5-71 6-8-71	3.54 x 10 ⁻⁴ (Not Req'd)	0.0 4.2 (N ₂ Only)	26.6 (Not Req'd)
U-1	0012	61A830248	N ₂	1/Vac	None	5-7-71	<2.63 x 10 ⁻⁶	0.0	23.0
U-1	0010	61A830248	N ₂	1/Vac 2/Vac	A51AE18	5-11-71 6-10-71	3.83 x 10 ⁻⁴ 6.0 x 10 ⁻⁶	3.2 5.2	19.3 20.0
U-1	0011	61A830248	N ₂	1/Vac	None	5-13-71	3.07 x 10 ⁻⁵	3.2	22.5
U-1	0007	61A830248	N ₂	1/Vac	None	5-18-71	3.56 x 10 ⁻⁵	2.4	24.8
U-1	0002	61A830248	N ₂	1/Vac 2/Amb 3/Vac	None A61AE14 None	6-4-71 6-7-71 7-9-71	1.23 x 10 ⁻⁴ 1.08 x 10 ⁻⁴ <1.09 x 10 ⁻⁶	4.0 Not Meas.	16.8 Not Meas. 19.1
U-1	P001	61A830243	O ₂	1/Vac	None	9-22-71	1.68 x 10 ⁻⁴	0.0	17.9
U-1	P006	61A830243	O ₂	1/Vac 2/Amb 3/Vac	A31AE33 New Valve Stem	9-29-71 10-7-71 3-31-71	1.6 x 10 ⁻⁴ (Not Req'd) 1.62 x 10 ⁻⁴	0.2 0.076 11.2	20.0 (Not Req'd) > 15.1
U-1	P003	61A830243	O ₂	1/Vac 2/Vac		10-4-71 4-12-72	8.72 x 10 ⁻⁶ 2.037 x 10 ⁻⁵	0.0 7.2	20.02 30.0
U-1	P005	61A830243	O ₂	1/Vac 2/Vac 3/Vac	None A42AE17 None	10-22-71 4-20-72 6-8-72	9.80 x 10 ⁻⁴ 1.209 x 10 ⁻³ 3.11 x 10 ⁻⁵	0.0 7.20 12.4	15.1 > 30.0 > 15.1
U-1	P004	61A830243	O ₂	1/Vac	None	10-28-71	<2.3 x 10 ⁻⁵	0.0	20.0
U-1	P002	61A830243	O ₂	1/Vac	None	11-30-71	<1.464 x 10 ⁻⁴	0.0	> 15.1
U-2	0009	61A830248	N ₂	1/Vac	None	11-2-71	2.88 x 10 ⁻⁴	2.1	19.4
U-2	0005	61A830248	N ₂	1/Vac 2/Vac	None None	12-8-71 12-8-71	1.602 x 10 ⁻³ 2.904 x 10 ⁻⁵	Not Meas. 0.0	Not Meas. > 15.1
U-2	0008	61A830248	N ₂	1/Vac	None	12-15-71	1.017 x 10 ⁻⁴	3.0	> 15.1
U-2	0013	61A830248	N ₂	1/Vac	None	1-26-72	1.293 x 10 ⁻⁴	8.4	> 15.1
U-2	P007	61A830243	O ₂	1/Vac	None	2-15-72	1.02 x 10 ⁻⁵	7.2	> 15.1
U-2	P008	61A830243	O ₂	1/Vac	None	2-17-72	4.665 x 10 ⁻⁶	12.0	> 15.1
U-2	0014	61A830248	N ₂	1/Vac	None	3-22-72	8.59 x 10 ⁻⁵	0.0	> 26.0
U-2	0004	61A830248	N ₂	1/Vac	None	4-27-72	2.7 x 10 ⁻⁵	4.0	> 15.1
U-2	P009	61A830243	O ₂	1/Vac	None	5-10-72	7.79 x 10 ⁻⁶	11.6	> 15.1
U-2	P010	61A830243	O ₂	1/Vac	None	5-18-72	1.11 x 10 ⁻⁴	12.0	> 30.0
U-2 U-1	0006	61A830248	N ₂	1/Vac	None	6-29-72	2.215 x 10 ⁻⁵	6.6	> 30.0
U-2	P011	61A830243	O ₂	1/Vac	None	7-6-72	2.38 x 10 ⁻⁵	13.2	> 30.0
U-2	P012	61A830243	O ₂	1/Vac	None	8-8-72	7.67 x 10 ⁻⁶	0.8	> 15.1
U-1 U-2	P013	61A830243	O ₂	1/Vac 2/Vac	A13AE12	1-19-73 3-20-73	1.91 x 10 ⁻⁵ 6.6 x 10 ⁻⁴	1.0 10.8	> 15.1 > 15.1

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